

DECLARATION UNDER 37 C.F.R. §1.132

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

SERIAL NO: 10/501,100

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FOR: NI BASED ALLOY WITH EXCELLENT CORROSION RESISTANCE TO
SUPERCRITICAL WATER ENVIRONMENTS CONTAINING INORGANIC ACIDS

EXAMINER: Jessee Roe

GROUP ART UNIT: 1793

DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS

ALEXANDRIA, VIRGINIA 22313-1450

SIR:

I, Katsuo Sugahara, a Japanese citizen residing at 2-1-3 Motomachi, Urawa-ku,
Saitama-shi, Saitama, 330-0073, declare and state that:

I obtained my doctor's degree in metallurgical engineering from Tokyo
Institute of Technology in March, 1998.

I entered Mitsubishi Materials Corporation in April 1 1990.

I am one of the Applicants of the above-identified application and I am well
familiar with the present case.

I have a good knowledge of the English language and have read and
understood the application papers and the prosecution history of this and the antecedent
applications as well as the Examiner's references cited therein.

PURPOSE, METHOD AND RESULTS

I. Purpose of the experiments:

The purpose of the Experiments described in this declaration is to show that it is indispensable all of Cr, Fe and Si are within the range of claim 2 in order to achieve the present invention, and that, if any of Cr, Fe and Si is not within the range of claim 2, the corrosion resistance deteriorates in supercritical water environments containing inorganic acids.

II. Method of the Experiments:

Using a raw material with a low C content in each case, the raw material was melted and cast in a normal high frequency induction furnace to prepare an ingot of thickness 12mm. The ingot was then subjected to homogenizing heat treatment for 10 hours at 1230°C. Subsequently, with the temperature held within a range from 1000°C to 1230°C, hot rolling was used to reduce the thickness by 1 mm per repetition, and this process was repeated until a final thickness of 5 mm was achieved. The sample was then subjected to solution treatment by holding the sample at 1200°C for 30 minutes followed by water quenching. The surface of the sample was then buffed, yielding a Ni based alloy sheet "Comparison 1 to 4".

Each of the Ni based alloy sheets "Comparison 1 to 4" was cut to prepare solution test specimens of dimensions 10 mm x 50 mm. In addition, in order to evaluate the effect of the phase stability on the corrosion resistance relative to a supercritical water environment containing inorganic acids, each of the Ni base alloy sheets "Comparison 1 to 4" was subjected to aging treatment by holding the sheet at 550°C for 1000 hours, and the sheet was then cut to prepare aged test specimens of dimensions 10 mm x 50 mm.

Next, a flow type corrosion test apparatus was prepared using a Hastelloy C-276 pipe as an autoclave. A test solution is pumped into one end of the Hastelloy C-276 pipe of this flow type corrosion test apparatus using a high pressure pump, and is discharged from the other end of the pipe, while the test solution inside the Hastelloy C-276 pipe is maintained at a predetermined flow rate. The test solution is heated by a heater provided on the Hastelloy C-276 pipe, and the test solution is able to be maintained at a predetermined temperature. In addition, the test solution discharged from the other end of the Hastelloy C-276 pipe of the flow type corrosion test apparatus passes through a pressure reducing valve and is recovered in a reservoir tank.

Using the flow type corrosion test apparatus described above, corrosion tests were conducted using the inorganic acid containing supercritical water simulated solutions described below.

(Aa) A test solution was prepared by mixing 0.2 mol/kg of sulfuric acid and 0.2 mol/kg of phosphoric acid into supercritical water with a fluid temperature of 550°C, a pressure of 40 MPa and a dissolved oxygen level of 8 ppm. This solution is an estimation of the supercritical water solution generated when VX gas is decomposed and oxidized in supercritical water (and is hereafter referred to as a simulated VX gas decomposition supercritical water solution). This simulated VX gas decomposition supercritical water solution was fed into the Hastelloy C-276 pipe of the aforementioned flow type corrosion test apparatus, and the flow rate of the simulated VX gas decomposition supercritical water solution inside the Hastelloy C-276 pipe was adjusted to 6 g/min, thus forming a supercritical water environment containing inorganic acids. Solution test specimens of the Ni based alloy sheets "Comparison 1 to 4" were then each held in this supercritical water environment for a period of 100 hours. The reduction in weight of the solution test specimen over the course of the test was divided by the surface area of the specimen to determine the weight loss per unit area for each test specimen.

In addition, in order to evaluate the effect of the phase stability on the corrosion resistance relative to a supercritical water environment containing inorganic acids, aged test specimens of the Ni based alloy sheets "Comparison 1 to 4" were each held in the above supercritical water environment containing inorganic acids for a period of 100 hours. The reduction in weight of the test specimen to determine the weight loss per unit area for each test specimen.

(Ab) A test solution was prepared by mixing 0.4 mol/kg of phosphoric acid and 0.1 mol/kg of hydrofluoric acid into supercritical water with a fluid temperature of 550°C, a pressure of 40 MPa and a dissolved oxygen level of 8 ppm. This solution is an estimation of the supercritical water solution generated when GB (sarin) gas is decomposed and oxidized in supercritical water (and is hereafter referred to as a simulated GB gas decomposition supercritical water solution). The simulated GB gas decomposition supercritical water solution was fed into the Hastelloy C-276 pipe of the aforementioned flow type corrosion test apparatus, and the flow rate of the simulated GB gas decomposition supercritical water solution inside the Hastelloy C-276 pipe was adjusted to 6 g/min, thus forming a supercritical water environment containing

inorganic acids. Solution test specimens of the Ni based alloy sheets "Comparison 1 to 4" were then each held in this supercritical water environment for a period of 100 hours. The reduction in weight of the solution test specimen over the course of the test was divided by the surface area of the specimens to determine the weight loss per unit area for each test specimen.

In addition, in order to evaluate the effect of the phase stability on the corrosion resistance relative to a supercritical water environment containing inorganic acids, aged test specimens of the Ni based alloy sheets "Comparison 1 to 4" were each held in the above supercritical water environment containing inorganic acids for a period of 100 hours. The reduction in weight of the test specimen over the course of the test was divided by the surface area of the aged test specimen to determine the weight loss per unit area for each test specimen.

III. Results:

Table 1

Ni based alloy sheet	Composition (% by weight)									Corrosion tests using simulated VX gas decomposition supercritical water solution		Corrosion tests using simulated GB gas decomposition supercritical water solution	
	Cr	Mo	Mg	N	Mn	Fe	Si	C#	Ni and unavoidable impurities	weight reduction in solution test specimen (mg/cm ²)	weight reduction in aged test specimen (mg/cm ²)	weight reduction in solution test specimen (mg/cm ²)	weight reduction in aged test specimen (mg/cm ²)
Comparison 1	44.5	0.80	0.07	0.018	0.15	-	0.33	0.02	remainder	13	35	16	33
Comparison 2	44.5	0.78	0.08	0.008	0.18	-	-	0.02	remainder	22	25	29	32
Comparison 3	44.5	0.61	0.07	0.009	0.11	-	-	0.02	remainder	19	23	21	24
Comparison 4	44.5	0.51	0.08	0.015	0.22	-	-	0.02	remainder	24	35	32	41

* indicates a value outside the composition range of the present invention (in gray background color)

The compositions (% by weight) of Comparison 1 to 4 are shown in Table 1. Please pay attention to the components (Cr, Fe and Si) marked by up-arrows in the Comparison 1 to 4.

(1) The result of Comparison 1 :

In Comparison 1, the content of Si is 0.33% by weight, which is not within the range of claim 2 (0.01 to 0.1%), but within the range of Kazuo Yamanaka et al. (from

some extent to 1%, see Examples), while the contents of other components are within the range of claim 2.

The result indicates that the content of Si beyond the range of claim 2, that is, quantities exceeding 0.1% results in an undesirable deterioration in the corrosion resistance relative to supercritical water environments containing inorganic acids.

(2) The result of Comparison 2:

In Comparison 2, the content of Cr is 39.8% by weight, which is not within the range of claim 2 (more than 43% to 50% or less), but within the range of Kazuo Yamanaka et al. (38 to 45%), while the contents of other components are within the range of claim 2.

The result indicates that, if the content of Cr is beyond the range of claim 2, that is, if quantities of Cr is 43% or less, the corrosion resistant effect cannot be achieved.

(3) The result of Comparison 3:

In Comparison 3, the content of Fe is 3.1% by weight, which is not within the range of claim 2 (0.05 to 1%), but within the range of Kazuo Yamanaka et al. (greater than 0 to 21.5 according to the Examiner's opinion), while the contents of other components are within the range of claim 2.

The result indicate that the content of Fe beyond the range of claim 2, that is, quantities exceeding 1% result in an undesirable deterioration in the corrosion resistance relative to supercritical water environments containing inorganic acids.

(4) The result of Comparison 4:

In Comparison 4, the contents of Cr, Fe and Si are not within the range of the present invention, but within the range of Kazuo Yamanaka et al., while the contents of other components are within the range of the present invention.

In the case that all of Cr, Fe and Si are not within the range of claim 2, the corrosion resistance in supercritical water environments containing inorganic acids became much worse than in the case of Comparison 1 to 3. Therefore, it is needed that all of Cr, Fe and Si are within the range of claim 2 in order to achieve the present invention.

IV. Conclusion:

For these reason, it can be fairly concluded that the present invention has a distinguished effect over Kazuo Yamanaka et al. by containing Cr, Fe and Si within the range of claim 2, not the range of Kazuo Yamanaka et al. and, if even one of the components is beyond the range of claim 2, the present invention cannot be achieved.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Katsuo Sugahara

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